

**TRANSMITTAL LETTER TO THE UNITED STATES  
DESIGNATED/ELECTED OFFICE (DO/EO/US)  
CONCERNING A FILING UNDER 35 U.S.C. 371**

7129

U.S. APPLICATION NO. (if known, see 37 CFR 1.5)

097674051

INTERNATIONAL APPLICATION NO.  
PCT/IB99/00708

INTERNATIONAL FILING DATE  
21 April 1999

PRIORITY DATE CLAIMED  
27 April 1998

**TITLE OF INVENTION**

**PROCESS FOR MAKING A READILY WATER-DISPERSIBLE NON-PARTICULATE DETERGENT PRODUCT  
FROM LOW-DENSITY PARTICULATE DETERGENT COMPOSITIONS**

**APPLICANT(S) FOR DO/EO/US**

ANGELL, Adrian John Waynforth; ZORB, Les Charles; STONE, Thomas Edward; ;

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information.

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☐ This express request to begin national examination procedures (35 U.S.C. 371(f) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(l).
4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. ☒ A copy of the International Application was filed (35 U.S.C. 371(c)(2))
  - a. ☐ is transmitted herewith (required only if not transmitted by the International Bureau).
  - b. ☒ has been transmitted by the International Bureau.
  - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☐ A translation of the International Application into English (35 U.S.C. 371(c)(2)).
7. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
  - a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
  - b. ☐ have been transmitted by the International Bureau.
  - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
  - d. ☒ have not been made and will not be made.
8. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☒ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
10. ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11. to 16. below concern document(s) or information included:

11. ☐ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☒ A **FIRST** preliminary amendment.  
☐ A **SECOND** or **SUBSEQUENT** preliminary amendment.
14. ☐ A substitute specification.
15. ☒ A change of power of attorney and/or address letter.
16. ☐ Other items or information:

"Express Mail" mailing label number

Date of Deposit

I hereby certify that this paper/fee is being deposited with the United States Postal Service "Express Mail Post Office to Addressee" service under 37 CFR 1.10 on the date indicated above and is addressed to The Assistant Commissioner of Patents, Washington, D.C. 20231

Administrative Mailing Application

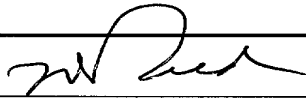
Signature

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FL15322527645  
25 October 2000

*[Signature]*

U.S. APPLICATION NO. (if known, see 37 CFR 1.5) <b>09/ 674051</b>		INTERNATIONAL APPLICATION NO. PCT/IB99/00708		ATTORNEY'S DOCKET NUMBER 7129	
				CALCULATIONS PTO USE ONLY	
ENTER APPROPRIATE BASIC FEE AMOUNT =				\$670	
Surcharge of \$130.00 for furnishing the oath or declaration later than [ ] 20 [ ] 30 months from the earliest claimed priority date (37 CFR 1.492(e)).				\$0	
CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE		
Total Claims	26-20 =	6	x \$22.00	\$132	
Independent Claims	3-3 =	0	x \$78.00	\$0	
MULTIPLE DEPENDENT CLAIM(S) (if applicable)			\$250.00	\$0	
TOTAL OF ABOVE CALCULATIONS =				\$802	
Processing fee of \$130.00 for furnishing the English translation later than [ ] 20 [ ] 30 months from the earliest claimed priority date (37 CFR 1.492(f)).				\$0	
TOTAL NATIONAL FEE =				\$802	
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28,3.31). \$40.00 per property +				\$0	
TOTAL FEES ENCLOSED =				\$802	
				Amount to be refunded	\$
				charged	\$
<p>a. [ ] A check in the amount of \$ ____ to cover the above fees is enclosed.</p> <p>b. [ x ] Please charge my Deposit Account No. <u>16-2480</u> in the amount of \$ <u>802</u> to cover the above fees. A duplicate copy of this sheet is enclosed.</p> <p>c. [ x ] The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. <u>16-2480</u>. A duplicate copy of this sheet is enclosed.</p> <p>NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.</p> <p>SEND ALL CORRESPONDENCE TO:</p>					
B.M. Bolam Patent Division The Procter & Gamble Company Ivorydale Technical Center 5299 Spring Grove Avenue Cincinnati, OH 45217				 Signature <u>T. David Reed</u> Name <u>32,931</u> Registration Number	

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## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In the matter of :  
 :  
 U.S. National Phase Entry :  
 Under 35 USC 371 from :  
 the International Application of :  
 ANGELL, Adrian John Waynforth et al :  
 Int'l Application No. PCT/IB99/00708 :  
 Filed in the RO/US on 21 April 1999 :  
 Entitled: Water-Dispersible Non-Particulate Detergent :  
 Product From Low-Density Particulate :  
 Detergent Particles :

PRELIMINARY AMENDMENT

Assistant Commissioner for Patents  
 Box PCT  
 Washington, D.C. 20231

Dear Sir:

Before computing the fees for entering the captioned International Application into the U.S. National Phase, please enter the following amendments **IN THE CLAIMS**:

- \* Please cancel Claims 3-14.

Please add the following new claims:

15. The process of claim 1 wherein said low density detergent particles of step (a) have a bulk density no less than about 400 g/l.

16. The process of claim 15 wherein said low density detergent particles of step (a) have a bulk density in a range of from about 400g/l to about 850g/l.

17. The process of claim 16 wherein said low density detergent particles of step (a) have a bulk density of about 700g/l.

18. The process of claim 1 wherein said liquid is a nonionic surfactant and is added to said low density detergent particles in an amount sufficient to reduce said intra-particle porosity by at least about 20%.

19. The process of claim 1 wherein said liquid is added in an amount in a range of from about 0.1% to about 10% by weight of said low density detergent particles.

20. The process of claim 1 wherein said liquid is selected from the group consisting of nonionic surfactants, anionic surfactants, water, polyethylene glycol, and mixtures thereof.

21. The process of claim 1 wherein said liquid is a nonionic surfactant.
22. The process of claim 1 wherein said liquid is polyethylene glycol having a molecular weight less than about 1000.
23. The process of claim 1 wherein said water-dispersible, non-particulate detergent product has at least about 5% greater dispersability in water as compared to a non-particulate detergent product having a density of at least about 1000 g/l but not having said liquid added to said low density particulate detergent composition per step (b).
24. The process of claim 23 wherein said water-dispersible, non-particulate detergent product has in a range of from about 5% to about 200% greater dispersability in water and wherein said liquid is polyethylene glycol having a molecular weight less than 1000.
25. The process of claim 1 wherein said liquid is a non-ionic liquid surfactant which is homogeneously mixed with said low density detergent particles.
26. A rapidly water-dispersible non-particulate detergent product formed by a process comprising the steps of:
- (a) providing low density detergent particles having an intra-particle porosity in a range of from about 5% to about 90% by volume;
  - (b) adding a liquid to said low density detergent particles in an amount sufficient to reduce said intra-particle porosity by at least about 10%; and
  - (c) compacting said low density detergent particles having said reduced intra-particle porosity, by applying a pressure in an amount sufficient to form said water-dispersible, non-particulate detergent product having a density of at least about 1000 g/l.
27. The detergent product formed by the process of claim 26 wherein said low density detergent particles of step (a) have an intra-particle porosity in a range of from about 10% to about 80% by volume.
28. The detergent product formed by the process of claim 26 wherein said low density detergent particles of step (a) have a bulk density no less than about 400 g/l.
29. The detergent product formed by the process of claim 28 wherein said low density detergent particles of step (a) have a bulk density in a range of from about 400g/l to about 850g/l.

30. The detergent product formed by the process of claim 26 wherein said liquid is a nonionic surfactant and is added to said low density detergent particles in an amount sufficient to reduce said intra-particle porosity by at least about 20%.
31. The detergent product formed by the process of claim 26 wherein said liquid is selected from the group consisting of nonionic surfactants, anionic surfactants, water, polyethylene glycol, and mixtures thereof.
32. The detergent product formed by the process of claim 26 wherein said liquid is polyethylene glycol having a molecular weight less than about 1000.
33. The detergent product formed by the process of claim 26 wherein said water-dispersible, non-particulate detergent product has at least about 5% greater dispersability in water as compared to a non-particulate detergent product having a density of at least about 1000 g/l but not having said liquid added to said low density particulate detergent composition per step (b).
34. The detergent product formed by the process of claim 26 wherein said liquid is polyethylene glycol having a molecular weight less than about 1000, said polyethylene glycol is homogeneously mixed with said low density detergent particles, and at least 80% by weight of said detergent product is dispersible in water after being immersed in water for no greater than about 3 minutes.
35. A method of laundering soiled clothes comprising the step of immersing said soiled clothes in an aqueous medium containing an effective amount of a non-particulate detergent product made by a process according to claim 1.
36. A process for producing a water-dispersible, non-particulate detergent product from a low density detergent composition, comprising the steps of:
- (a) providing spray dried detergent granules having a bulk density less than about 650 g/l;
  - (b) providing agglomerated detergent particles having a bulk density in a range of from about 700 g/l to about 900 g/l;
  - (c) mixing said spray dried detergent granules and said agglomerated detergent particles to form a low density detergent composition, wherein said spray dried detergent granules are present in a range of from about 40% to about 80% by weight of said low density detergent composition and said agglomerated detergent particles are present in a range of from about 20% to about 60% by weight of said low density detergent composition;
  - (d) adding a liquid to said low density detergent composition in an amount sufficient to reduce said intra-particle porosity by at least about 10%; and
  - (e) compacting said low density detergent composition having said reduced intra-particle porosity, by applying a pressure in an amount sufficient to form said water-dispersible, non-particulate detergent product having a density of at least about 1000 g/l.

37. A method of laundering fabric materials in a washing machine, comprising the steps of:  
providing a flexible porous bag adapted for receiving a non-particulate detergent product;  
providing a non-particulate detergent product made by a process according to claim 1;  
placing said non-particulate detergent product within said flexible porous bag;  
placing said flexible porous bag containing said detergent product in said washing machine  
with said fabric materials to be washed; and

said flexible porous bag being adapted for permitting entry of an aqueous washing medium  
through said bag, thereby dissolving said non-particulate detergent product placed therein, into said  
aqueous washing medium, and releasing a resultant wash solution from inside of said bag to outside  
of said bag into said aqueous wash medium during a wash cycle.

38. The method of claim 37 wherein said non-particulate detergent product has a density of at  
least 1000 g/l.

The support for these amendments is found in the claims as originally filed. These amendments are  
being entered to bring the claims into conformance with, *inter alia*, 37 CFR §1.75; no new matter is  
added.

Respectfully submitted for Applicants,

By:



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WATER-DISPERSIBLE NON-PARTICULATE DETERGENT PRODUCT FROM LOW-DENSITY PARTICULATE DETERGENT PARTICLES

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TECHNICAL FIELD

The present invention relates to detergent compositions in non-particulate form. More particularly, the invention relates to a process for making a non-particulate detergent product, such as a tablet, block, or bar which sinks in water, readily disintegrates in water and is rapidly dispersible in water, by using a low density particulate detergent composition having a particulate bulk density less than 800 g/l as a starting material.

BACKGROUND OF THE INVENTION

Non-particulate detergents are an attractive alternative to granular or particulate forms of detergents from the standpoint of simplifying the dosing of such detergents for automatic laundry or dishwashing machines. Non-particulate detergents are usually supplied in the form of bars, tablets or briquettes and they not only prevent spillage of the detergent composition but also eliminate the need for the consumer to estimate the correct dosage of the detergent composition per wash. Non-particulate detergents minimize the contact by the consumer with the detergent.

An important factor for successful performance of a non-particulate detergent is its ability to disintegrate and dissolve in the washing machine in a controlled manner according to a desired dissolution profile during the program cycle of the machine. Another important performance factor is that the non-particulate detergent should be hard enough to facilitate easy handling of the detergent prior to use, so that it does not inadvertently lose its structure, crumble, or deteriorate, both during the packaging, transport and storage and during handling by the end consumer prior to actual use.

Additionally, a very desirable feature of a non-particulate detergent, such as for example, a tablet, is its ability to sink in water and rapidly disperse in water to

form a wash solution. In order to sink in water, a detergent tablet must have a density greater than 1000 g/l and in order to disperse in water, a detergent tablet must be able to break up in water. However, when laundry tablets are made from low bulk density detergents, such as those made by spray dried processes, wherein the detergent powder has a bulk density less than about 650 g/l, the problem frequently encountered is that the force required to compact the detergent powder into tablets having a density of at least 1000 g/l is so high that the detergent tablets do not readily disperse in water. This problem is further escalated by the fact that detergent powders made from spray dried processes tend to be more porous and sticky. Thus when these detergent powders are pressed into tablets having a density of at least 1000 g/l, the powder particles stick together and consequently the tablet does not readily break up and dissolve in water. Conversely, if the tablets made from low bulk density detergent powders are compacted using a lower force, they generally disperse in water but at a slower rate because they have a density less than 1000 g/l and thus tend to float in water before fully dispersing in water.

The above problem is usually not encountered when making detergent tablets from a detergent powder made by agglomeration processes because detergent powders made by agglomeration processes usually have a bulk density in a range of about 700 g/l to about 850 g/l and consequently, the force required to compress the powder into a tablet having a density of at least 1000 g/l is not so high. Thus detergent tablets made by compacting detergent powders made from agglomeration process usually sink in water. However, agglomeration process detergents or "agglomerates", which inherently have higher density than spray dried process detergents or "spray dried granules", generally exhibit slower dissolution rates in water, as compared to spray dried granules.

Thus the production of detergent tablets is a complex matter. It involves more than the mere selection of components or the compression of a particular detergent composition into a tablet. The tablet must be capable of withstanding the shocks of packaging, handling and distribution without crumbling. In other words the tablet must be strong. But the tablet must also have a satisfactory rate of disintegration when immersed in water. The tablets known so far have generally



shown too long a disintegration time, in favor of their strength, or they have had a very low strength, in favor of their shorter disintegration time.

65 It is highly desirable to have a laundry detergent tablet with a core which is formed by compressing a particulate material which has a deterative surfactant and a builder and wherein the particulate material has a bulk density less than 850 g/l so that the detergent tablet formed by compressing such a particulate material not only sinks in water but also rapidly disintegrates and readily dissolves in water.

70 This kind of a tablet performance has heretofore not been available and this level of performance requires not only careful selection of the type of detergent that makes up the core, but also a carefully selected density range of the particulate detergent to improve the dissolution of the detergent. To achieve a particulate detergent bulk density within an optimal range, it is desirable to exploit not only the  
75 benefits of using mixtures of spray dried granules and agglomerates but also processing the detergent particles in a manner so as to reduce their inherent porosity and bring that particle porosity within an optimally desired range, prior to the step of compacting that detergent particles into a non-particulate form. It is thus necessary to carefully tailor the particle porosity of the particulate detergent prior to  
80 compaction. The present invention overcomes the problems as set forth above.

### BACKGROUND ART

The prior art is replete with methods of forming tablets and coating tablets.

One approach has been to use acetate salt to improve the dissolution rate of detergents compressed in the form of tablets. EP-A-0002293, published on 13<sup>th</sup> June  
85 1979, discloses detergent tablets containing hydrated salt. The preferred hydrate salt is a mixture of sodium acetate trihydrate and sodium metaborate tetrahydrate.

Another approach known in the art is to use effervescent aids to improve tablet disintegration. CA-A-2040307 discloses laundry detergent tablets comprising anionic surfactants mixed with sodium carbonate and citric acid.

90 As far as coated tablets are concerned, GB-A-0 989 683, published on 22nd April 1965, discloses a process for preparing a particulate detergent from surfactants and inorganic salts; spraying on water-soluble silicate; and pressing the detergent particles into a solid form-retaining tablet. Finally a readily water-soluble organic

film-forming polymer (for example, polyvinyl alcohol) provides a coating to make  
95 the detergent tablet resistant to abrasion and accidental breakage.

European publication, EP-A-0 002 293, published on 13th June 1979,  
discloses a tablet coating comprising hydrated salt such as acetate, metaborate,  
orthophosphate, tartrate, and sulphate. Another European publication, EP-A-0 716  
144, published on 12th June 1996, also discloses laundry detergent tablets with  
100 water-soluble coatings which may be organic polymers including acrylic/maleic co-  
polymer, polyethylene glycol, PVPVA, and sugar.

U.S. Patent No. 5,658,874, issued on Aug. 19, 1997 to Alan P. Davies et al.  
and assigned to Lever Brothers Company, discloses a process for making tablets of a  
detergent composition comprising detergent active compound, detergency builder  
105 and other ingredients, in which at least some particles of the composition are  
individually coated with a material that functions as a binder and a disintegrant  
capable, when the tablet is immersed in water, for disrupting the structure of the  
tablet. However, this patent discloses the use of high molecular weight binders that  
generally lead to tablets that are more porous.

#### 110 SUMMARY OF THE INVENTION

The invention meets the needs above by providing a process for producing a  
water-dispersible, non-particulate detergent product. Specifically, in one aspect of  
the present invention, the process includes the step of providing a low density  
particulate detergent composition having an intra-particle porosity in a range of from  
115 about 10% to about 90% by volume. The process further includes the step of adding  
a liquid to the low density particulate detergent composition in an amount sufficient  
to reduce the intra-particle porosity by at least about 10%. The process then includes  
the step of compacting the low density particulate detergent composition having  
reduced intra-particle porosity by applying a pressure in an amount sufficient to  
120 form the water-dispersible non-particulate detergent product having a density of at  
least about 1000 g/l.

In another aspect of the present invention, a rapidly water-dispersible non-  
particulate detergent product is disclosed. The product is formed by a process  
comprising the steps of providing low density detergent particles having an intra-

125 particle porosity in a range of from about 5% to about 90% by volume, adding a  
liquid to the low density detergent particles in an amount sufficient to reduce the  
intra-particle porosity by at least about 10%, and compacting the low density  
detergent particles having the reduced intra-particle porosity, by applying a pressure  
in an amount sufficient to form the water-dispersible, non-particulate detergent  
130 product having a density of at least about 1000 g/l.

In still another aspect of the present invention, a method of laundering soiled  
clothes includes the step of immersing said soiled clothes in an aqueous medium  
containing an effective amount of a non-particulate detergent product made by a  
process as set forth above.

135 In yet another aspect of the present invention, a process for producing a  
water-dispersible, non-particulate detergent product from a low density detergent  
composition is disclosed. The process includes the steps of providing spray dried  
detergent granules having a bulk density less than about 650 g/l. The process further  
include the step of providing agglomerated detergent particles having a bulk density  
140 in a range of from about 700 g/l to about 900 g/l. The process still further includes  
the steps of mixing the spray dried detergent granules and the agglomerated  
detergent particles to form a low density detergent composition, wherein the spray  
dried detergent granules are present in a range of from about 40% to about 80% by  
weight of the low density detergent composition and the agglomerated detergent  
145 particles are present in a range of from about 20% to about 60% by weight of the  
low density detergent composition. The process also includes the step of adding a  
liquid to the low density detergent composition in an amount sufficient to reduce  
said intra-particle porosity by at least about 10%, and compacting the low density  
detergent composition having the aforementioned reduced intra-particle porosity, by  
150 applying a pressure in an amount sufficient to form the water-dispersible, non-  
particulate detergent product having a density of at least about 1000 g/l.

In still another aspect of the present invention, a method of laundering fabric  
materials in a washing machine is provided. The method includes the steps of  
155 providing a flexible porous bag adapted for receiving a non-particulate detergent

product, providing a non-particulate detergent product made according to the process described above, placing the non-particulate detergent product within the flexible porous bag, and placing the flexible porous bag containing the detergent product in the washing machine with the fabric materials to be washed. The flexible porous bag is adapted for permitting entry of an aqueous washing medium through the bag, thereby dissolving the non-particulate detergent product placed therein, into the aqueous washing medium, and releasing a resultant wash solution from inside of the bag to outside of the bag and into the aqueous wash medium during a wash cycle.

### DETAILED DESCRIPTION OF THE INVENTION

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#### Process

In the preferred embodiment of the present invention, the process includes the step of providing a low density particulate detergent composition having an intra-particle porosity desirably, in a range of from about 10% to about 90% by volume. Preferably, the porosity is in a range of from about 15% to about 70% by volume and most preferably, the porosity is in a range of from about 20% to about 65% by volume. This porosity is measured by a Mercury Porosimeter, which technique is well known to those skilled in the art and need not be described in any detail here.

#### The particulate detergent composition

The term "particulate" as used herein means forms such as powders, granules, particles, flakes and other similar particulate forms that are capable of being compacted into a more dense non-particulate form.

In particular for laundry tablets, detergent particles having ingredients such as builder and surfactant can be spray-dried in a conventional manner and then compacted at a suitable pressure. The surfactants and builders normally provide a substantial part of the cleaning power of the tablet. The term "builder" is intended to mean all materials which tend to remove calcium ion from solution, either by ion exchange, complexation, sequestration or precipitation.

The particulate material used for making the detergent tablet provided in this invention can be made by any particulation or granulation process. An example of such a process is spray drying (in a co-current or counter current spray drying tower)

which typically gives "spray-dried" detergent granules having low bulk densities of 600g/l or lower. Particulate materials of higher density can be prepared by granulation and densification in a high shear batch mixer/granulator or by a continuous granulation and densification process (e.g. using Lodige® CB and/or Lodige® KM mixers). Other suitable processes include fluid bed processes, compaction processes (e.g. roll compaction), extrusion, as well as any particulate material made by any chemical process like flocculation, crystallization sentering, etc. The individual particles can also be in any other form, such as for example, particle, granule, sphere or grain.

The particulate materials may be mixed together by any conventional means, for example, a concrete mixer, Nauta mixer, ribbon mixer or any other. Alternatively the mixing process may be carried out continuously by metering each component by weight on to a moving belt, and blending them in one or more drum(s) or mixer(s). A liquid spray-on to the mix of particulate materials (e.g. non-ionic surfactants) may be carried out. Other liquid ingredients may also be sprayed on to the mix of particulate materials either separately or premixed. For example perfume and slurries of optical brighteners may be sprayed. A finely divided flow aid (dusting agent such as zeolites, carbonates, silicas) can be added to the particulate materials after spraying the non-ionic, preferably towards the end of the process, to make the mix less sticky.

The detergent particles can be made by an agglomerate process comprising the steps of:

- i) admixing one or more detergent surfactants, a perborate component and an acid source and optionally other detergent ingredients to form a mixture; and
- ii) agglomerating the mixture to form agglomerated particles or "agglomerates".

Typically, such an agglomeration process involves mixing an effective amount of powder, including the acid source, with a high active surfactant paste in one or more agglomerators such as a pan agglomerator, a Z-blade mixer or more preferably in-line mixers, preferably two, such as those manufactured by Schugi (Holland) BV, 29 Chroomstraat 8211 AS, Lelystad, Netherlands, and Gebruder

Lodige Maschinenbau GmbH, D-4790 Paderborn 1, Elsenerstrasse 7-9, Postfach 2050, Germany. Preferably a high shear mixer is used, such as a Lodige CB (Trade Name). Most preferably, a high shear mixer is used in combination with a low shear mixer, such as a Lodige CB (Trade Name) and a Lodige KM (Trade name) or Schugi KM (Trade Name). Optionally, only one or more low shear mixer are used. Preferably, the agglomerates are thereafter dried and/ or cooled.

Another agglomeration process involves mixing of various components of the final agglomerate in different stages, using an fluidized bed. For example, a preferred particulate detergent in accordance with the present invention can be agglomerated by addition, preferably by spraying on, of nonionic, anionic surfactants and optionally a wax, or mixtures thereof, to the acid source in powdered form and other optional ingredients. Then, additional components, including the perborate bleach and optionally the alkali source or part thereof, can be added and agglomerated in one or more stages, thus forming the final agglomerate particle.

The agglomerates may take the form of flakes, prills, marumes, noodles, ribbons, but preferably take the form of granules. A preferred way to process the particles is by agglomerating powders (e.g. aluminosilicate, carbonate) with high active surfactant pastes and to control the particle size of the resulting agglomerates within specified limits. Typical particle sizes are from 0.10 mm to 5.0 mm in diameter, preferably from 0.25 mm to 3.0 mm in diameter, most preferably from 0.40 mm to 1.00 mm in diameter. Typically, the "agglomerates" have a bulk density desirably ,of at least 700 g/l and preferably, in a range of from about 700 g/l to about 900 g/l.

A high active surfactant paste comprising a mix of, typically, from 50% by weight to 95% by weight, preferably 70% by weight to 85% by weight of surfactant, and optionally it can contain an appropriate acid source. The paste may be pumped into the agglomerator at a temperature high enough to maintain a pumpable viscosity, but low enough to avoid degradation of the anionic surfactants used. An operating temperature of the paste of 50°C to 80°C is typical. Such pastes and methods for making and processing such pastes is for example described in WO 93/03128. In an especially preferred embodiment of the present invention, the

detergent particles made by agglomeration process have a bulk density of greater  
250 than about 600 g/l and the detergent is in the form of powder or a granulate.

In the preferred embodiment of the present invention, the particulate  
detergent composition is a mixture of spray dried process and agglomeration process  
detergents, such that the final bulk density of the detergent composition is in a range  
of desirably, no greater than about 900 g/l, more desirably, in a range of from about  
255 600 g/l to about 850 g/l, and preferably, in a range of from about 625 g/l to about  
725 g/l. These ranges of bulk density are desirable because if the bulk density of the  
particulate detergent from which the tablet is to be compressed is greater than about  
900 g/l, then the solubility of the detergent tablet will be detrimentally affected. A  
bulk density less than about 600 g/l is undesirable because at values less than about  
260 600 g/l, the amount of pressure required to form a detergent tablet having a density  
of at least 1000 g/l is so high that the tablet will not break up easily in water and will  
not dissolve rapidly.

To achieve the desired bulk densities as set forth above, the particulate  
detergent composition contains selected amounts of spray dried granules and  
265 detergent agglomerates in an optimum proportion. In this regard, the composition  
comprises desirably from about 40% to about 80%, preferably from about 40% to  
about 60%, and more preferably from about 45% to about 55%, by weight, of spray  
dried. Desirably, the composition includes from about 20% to about 60%,  
preferably from about 40% to about 60%, and more preferably from about 45% to  
270 about 55%, by weight, of agglomerates.

#### Addition of Liquid to reduce porosity of detergent particles

In the preferred embodiment of the present invention, the process further  
includes the step of adding a liquid to the low density particulate detergent  
composition in an amount sufficient to reduce the intra-particle porosity by at least  
275 about 10%. It is desirable to reduce the intra-particle porosity by at least about 10%,  
and preferably by at least 20%, because if the porosity reduction is less than about  
10%, no appreciable dispersibility of the detergent tablet is expected. Without being  
bound to a specific theory, it is theorized that the porosity reduction of the spray  
dried granules and agglomerates, and the resultant increase in particle density,

280 causes the agglomerates to harden while the spray dried granules remain relatively  
softer. When a pressure is applied to compact the detergent particle mixture into a  
tablet, for example, there is believed to be some mechanical interlocking between  
the hard agglomerated and the softer spray dried granules. The spray dried granules  
are believed to "flow" into the interstitial spaces between the particles, thereby  
285 increasing the mechanical bonding between the particles upon the application of  
pressure. This results in the formation of a detergent tablet that is not only strong  
and can resist breakage, but also can disintegrate rapidly in water because the  
detergent particles do not "stick" together. Due to the mixture of spray dried  
granules and agglomerates in the composition, the dissolution rate of the detergent  
290 tablet is also increased because of the faster solubility of spray dried particles in  
water.

Any liquid for reducing porosity can be used to carry out the invention,  
however non-ionics are preferred as they also provide detergency benefits. Suitable  
nonionic surfactants include compounds produced by the condensation of alkylene  
oxide groups (hydrophilic in nature) with an organic hydrophobic compound, which  
295 may be aliphatic or alkyl aromatic in nature. The length of the polyoxyalkylene  
group which is condensed with any particular hydrophobic group can be readily  
adjusted to yield a water-soluble compound having the desired degree of balance  
between hydrophilic and hydrophobic elements. Other examples are nonionic  
300 surfactants such as the polyethylene oxide condensates of alkyl phenols, e.g., the  
condensation products of alkyl phenols having an alkyl group containing from about  
6 to 16 carbon atoms, in either a straight chain or branched chain configuration, with  
from about 4 to 25 moles of ethylene oxide per mole of alkyl phenol. Still other  
nonionics are the water-soluble condensation products of aliphatic alcohols  
305 containing from 8 to 22 carbon atoms, in either straight chain or branched  
configuration, with an average of up to 25 moles of ethylene oxide per mole of  
alcohol.

Polyhydroxy fatty acid amides may be produced by reacting a fatty acid ester  
and an N-alkyl polyhydroxy amine. The preferred amine for use in the present  
310 invention is N-(R1)--CH<sub>2</sub>(CH<sub>2</sub>OH)<sub>4</sub>--CH<sub>2</sub>--OH, where R1 is typically a alkyl, e.g.



methyle group; and the preferred ester is a C12-C20 fatty acid methyl ester. Methods of manufacturing polyhydroxy fatty acid amides have been described in WO 92 6073, published on 16th Apr., 1992. This application describes the preparation of polyhydroxy fatty acid amides in the presence of solvents. In a highly preferred embodiment of the invention N-methyl glucamine is reacted with a C12-C20 methyl ester. It also says that the formulator of granular detergent compositions may find it convenient to run the amidation reaction in the presence of solvents which comprise alkoxyated, especially ethoxyated (EO 3-8) C12-C14 alcohols (page 15, lines 22-27). This can directly yield nonionic surfactant systems which are preferred in the present invention, such as those comprising N-methyl glucosamide and C12-C14 alcohols with an average of 3 ethoxylate groups per molecule. Nonionic surfactant systems, and granular detergents made from such systems have been described in WO 92 6160, published on 16th Apr., 1992. This application describes (example 15) a granular detergent composition prepared by fine dispersion mixing in an Eirich RV02 mixer which comprises N-methyl glucosamide (10%), nonionic surfactant (10%). Both of these patent applications describe nonionic surfactant systems together with suitable manufacturing processes for their synthesis, which have been found to be suitable for use in the present invention.

Other nonionic surfactants which may be used as components of the surfactant systems herein include ethoxyated nonionic surfactants, glycerol ethers, glucosamides, glycerol amides, glycerol esters, fatty acids, fatty acid esters, fatty amides, alkyl polyglucosides, alkyl polyglycol ethers, polyethylene glycols, ethoxyated alkyl phenols and mixtures thereof.

In the preferred embodiment, the liquid used is one of ionic surfactants, anionic surfactants, water and polyethylene glycol, and mixtures thereof. Preferably, the liquid is a nonionic surfactant, and more preferably, it is polyethylene glycol (PEG). Most preferably, the PEG has a molecular weight of less than about 1000 and in the best mode, the molecular weight of PEG is 200. In the preferred embodiment, PEG is added to the detergent composition in a range of from about 0.1% to about 10% by weight and preferably about 5% by weight. In the preferred

embodiment, the addition of PEG having a molecular weight of about 200 results in a porosity reduction in a range of 5% to 200%.

#### Dry Detergent Material

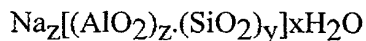
The starting dry detergent material of the present process preferably comprises materials selected from the group consisting of carbonates, sulfates, carbonate/sulfate complexes, tripolyphosphates, tetrasodium pyrophosphate, citrates, aluminosilicates, cellulose-based materials and organic synthetic polymeric absorbent gelling materials. More preferably, the dry detergent material is selected from the group consisting of aluminosilicates, carbonates, sulfates, carbonate/sulfate complexes, and mixtures thereof. Most preferably, the dry detergent material comprise a detergent aluminosilicate builder which are referenced as aluminosilicate ion exchange materials and sodium carbonate.

The aluminosilicate ion exchange materials used herein as a detergent builder preferably have both a high calcium ion exchange capacity and a high exchange rate. Without intending to be limited by theory, it is believed that such high calcium ion exchange rate and capacity are a function of several interrelated factors which derive from the method by which the aluminosilicate ion exchange material is produced. In that regard, the aluminosilicate ion exchange materials used herein are preferably produced in accordance with Corkill et al, U.S. Patent No. 4,605,509 (Procter & Gamble), the disclosure of which is incorporated herein by reference.

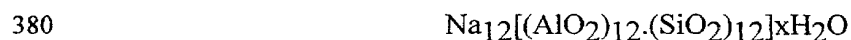
Preferably, the aluminosilicate ion exchange material is in "sodium" form since the potassium and hydrogen forms of the instant aluminosilicate do not exhibit the as high of an exchange rate and capacity as provided by the sodium form. Additionally, the aluminosilicate ion exchange material preferably is in over dried form so as to facilitate production of crisp detergent agglomerates as described herein. The aluminosilicate ion exchange materials used herein preferably have particle size diameters which optimize their effectiveness as detergent builders. The term "particle size diameter" as used herein represents the average particle size diameter of a given aluminosilicate ion exchange material as determined by conventional analytical techniques, such as microscopic determination and scanning electron microscope (SEM). The preferred particle size diameter of the aluminosilicate is from about 0.1

micron to about 10 microns, more preferably from about 0.5 microns to about 9 microns. Most preferably, the particle size diameter is from about 1 microns to about 8 microns.

375 Preferably, the aluminosilicate ion exchange material has the formula



wherein z and y are integers of at least 6, the molar ratio of z to y is from about 1 to about 5 and x is from about 10 to about 264. More preferably, the aluminosilicate has the formula



wherein x is from about 20 to about 30, preferably about 27. These preferred aluminosilicates are available commercially, for example under designations Zeolite A, Zeolite B and Zeolite X. Alternatively, naturally-occurring or synthetically derived aluminosilicate ion exchange materials suitable for use herein can be made as described in Krummel et al, U.S. Patent No. 3,985,669, the disclosure of which is incorporated herein by reference.

The aluminosilicates used herein are further characterized by their ion exchange capacity which is at least about 200 mg equivalent of  $\text{CaCO}_3$  hardness/gram, calculated on an anhydrous basis, and which is preferably in a range from about 300 to 352 mg equivalent of  $\text{CaCO}_3$  hardness/gram. Additionally, the instant aluminosilicate ion exchange materials are still further characterized by their calcium ion exchange rate which is at least about 2 grains  $\text{Ca}^{++}$ /gallon/minute/-gram/gallon, and more preferably in a range from about 2 grains  $\text{Ca}^{++}$ /gallon/minute/-gram/gallon to about 6 grains  $\text{Ca}^{++}$ /gallon/minute/-gram/gallon.

395 Additionally, those builder materials discussed previously as an optional coating agent can be used herein. These particular builder materials have the formula  $(\text{M}_x)_i \text{Ca}_y (\text{CO}_3)_z$  wherein x and i are integers from 1 to 15, y is an integer from 1 to 10, z is an integer from 2 to 25,  $\text{M}_i$  are cations, at least one of which is a water-soluble, and the equation  $\sum_i = 1-15(x_i \text{ multiplied by the valence of } \text{M}_i) + 2y = 2z$  is satisfied such that

400 the formula has a neutral or "balanced" charge. Additional details and examples of these builder materials have been set forth previously and are incorporated herein by

reference. Preferably, these builder materials are selected from the group consisting of  $\text{Na}_2\text{Ca}(\text{CO}_3)_2$ ,  $\text{K}_2\text{Ca}(\text{CO}_3)_2$ ,  $\text{Na}_2\text{Ca}_2(\text{CO}_3)_3$ ,  $\text{NaKCa}(\text{CO}_3)_2$ ,  $\text{NaKCa}_2(\text{CO}_3)_3$ ,  $\text{K}_2\text{Ca}_2(\text{CO}_3)_3$ , and combinations thereof.

405

#### Adjunct Detergent Ingredients

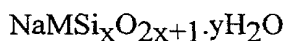
The starting dry detergent material in the present process can include additional detergent ingredients and/or, any number of additional ingredients can be incorporated in the detergent composition during subsequent steps of the present process. These adjunct ingredients include other detergency builders, bleaches, bleach activators, suds  
410 boosters or suds suppressers, anti-tarnish and anticorrosion agents, soil suspending agents, soil release agents, germicides, pH adjusting agents, non-builder alkalinity sources, chelating agents, smectite clays, enzymes, enzyme-stabilizing agents and perfumes. See U.S. Patent 3,936,537, issued February 3, 1976 to Baskerville, Jr. et al., incorporated herein by reference.

415

Other builders can be generally selected from the various water-soluble, alkali metal, ammonium or substituted ammonium phosphates, polyphosphates, phosphonates, polyphosphonates, carbonates, borates, polyhydroxy sulfonates, polyacetates, carboxylates, and polycarboxylates. Preferred are the alkali metal, especially sodium, salts of the above. Preferred for use herein are the phosphates,  
420 carbonates,  $\text{C}_{10-18}$  fatty acids, polycarboxylates, and mixtures thereof. More preferred are sodium tripolyphosphate, tetrasodium pyrophosphate, citrate, tartrate mono- and di-succinates, and mixtures thereof (see below).

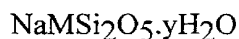
In comparison with amorphous sodium silicates, crystalline layered sodium silicates exhibit a clearly increased calcium and magnesium ion exchange capacity. In  
425 addition, the layered sodium silicates prefer magnesium ions over calcium ions, a feature necessary to insure that substantially all of the "hardness" is removed from the wash water. These crystalline layered sodium silicates, however, are generally more expensive than amorphous silicates as well as other builders. Accordingly, in order to provide an economically feasible laundry detergent, the proportion of crystalline  
430 layered sodium silicates used must be determined judiciously.

The crystalline layered sodium silicates suitable for use herein preferably have the formula



wherein M is sodium or hydrogen, x is from about 1.9 to about 4 and y is from about 0

435 to about 20. More preferably, the crystalline layered sodium silicate has the formula



wherein M is sodium or hydrogen, and y is from about 0 to about 20. These and other crystalline layered sodium silicates are discussed in Corkill et al, U.S. Patent No.

4,605,509, previously incorporated herein by reference.

440 Specific examples of inorganic phosphate builders are sodium and potassium tripolyphosphate, pyrophosphate, polymeric metaphosphate having a degree of polymerization of from about 6 to 21, and orthophosphates. Examples of polyphosphonate builders are the sodium and potassium salts of ethylene diphosphonic acid, the sodium and potassium salts of ethane 1-hydroxy-1,  
445 1-diphosphonic acid and the sodium and potassium salts of ethane, 1,1,2-triphosphonic acid. Other phosphorus builder compounds are disclosed in U.S. Patents 3,159,581; 3,213,030; 3,422,021; 3,422,137; 3,400,176 and 3,400,148, all of which are incorporated herein by reference.

Examples of nonphosphorus, inorganic builders are tetraborate decahydrate  
450 and silicates having a weight ratio of  $\text{SiO}_2$  to alkali metal oxide of from about 0.5 to about 4.0, preferably from about 1.0 to about 2.4. Water-soluble, nonphosphorus organic builders useful herein include the various alkali metal, ammonium and substituted ammonium polyacetates, carboxylates, polycarboxylates and polyhydroxy sulfonates. Examples of polyacetate and polycarboxylate builders are  
455 the sodium, potassium, lithium, ammonium and substituted ammonium salts of ethylene diamine tetraacetic acid, nitrilotriacetic acid, oxydisuccinic acid, mellitic acid, benzene polycarboxylic acids, and citric acid.

Polymeric polycarboxylate builders are set forth in U.S. Patent 3,308,067, Diehl, issued March 7, 1967, the disclosure of which is incorporated herein by  
460 reference. Such materials include the water-soluble salts of homo- and copolymers of aliphatic carboxylic acids such as maleic acid, itaconic acid, mesaconic acid, fumaric acid, aconitic acid, citraconic acid and methylene malonic acid. Some of

these materials are useful as the water-soluble anionic polymer as hereinafter described, but only if in intimate admixture with the non-soap anionic surfactant.

465 Other suitable polycarboxylates for use herein are the polyacetal carboxylates described in U.S. Patent 4,144,226, issued March 13, 1979 to Crutchfield et al, and U.S. Patent 4,246,495, issued March 27, 1979 to Crutchfield et al, both of which are incorporated herein by reference. These polyacetal carboxylates can be prepared by bringing together under polymerization conditions an ester of glyoxylic acid and a  
470 polymerization initiator. The resulting polyacetal carboxylate ester is then attached to chemically stable end groups to stabilize the polyacetal carboxylate against rapid depolymerization in alkaline solution, converted to the corresponding salt, and added to a detergent composition. Particularly preferred polycarboxylate builders are the ether carboxylate builder compositions comprising a combination of tartrate  
475 monosuccinate and tartrate disuccinate described in U.S. Patent 4,663,071, Bush et al., issued May 5, 1987, the disclosure of which is incorporated herein by reference.

Bleaching agents and activators are described in U.S. Patent 4,412,934, Chung et al., issued November 1, 1983, and in U.S. Patent 4,483,781, Hartman, issued November 20, 1984, both of which are incorporated herein by reference. Chelating  
480 agents are also described in U.S. Patent 4,663,071, Bush et al., from Column 17, line 54 through Column 18, line 68, incorporated herein by reference. Suds modifiers are also optional ingredients and are described in U.S. Patents 3,933,672, issued January 20, 1976 to Bartoletta et al., and 4,136,045, issued January 23, 1979 to Gault et al., both incorporated herein by reference.

485 Suitable smectite clays for use herein are described in U.S. Patent 4,762,645, Tucker et al, issued August 9, 1988, Column 6, line 3 through Column 7, line 24, incorporated herein by reference. Suitable additional detergency builders for use herein are enumerated in the Baskerville patent, Column 13, line 54 through Column 16, line 16, and in U.S. Patent 4,663,071, Bush et al, issued May 5, 1987, both incorporated  
490 herein by reference.

The non-particulate detergent product

The detergent tablets can be prepared simply by mixing the solid ingredients together and compressing the mixture in a conventional tablet press as used, for example, in the pharmaceutical industry.

495 The detergent tablets provided can be made in any size or shape. Prior to compaction, the detergent particles may be surface treated with a flow aid according to the present invention. The detergent tablets provided may be manufactured by using any compacting process, such as tableting, briquetting, or extrusion, preferably tableting. Suitable equipment includes a standard single stroke or a  
500 rotary press (such as Courtoy®, Korch®, Manesty®, or Bonals®). As used herein, the term "non-particulate detergent product" includes physical shapes such as tablets, blocks, bars and the like.

#### Coating for non-particulate detergent product

In one embodiment, the tablets are coated with a coating in order to provide  
505 mechanical strength and shock and chip resistance to the compressed tablet core. The tablets are coated with a coating that is substantially insoluble in water so that the tablet does not absorb moisture, or absorbs moisture at only a very slow rate. The coating is strong so that moderate mechanical shocks to which the tablets are subjected during handling, packing and shipping result in no more than very low  
510 levels of breakage or attrition. Further, the coating is preferably brittle so that the tablet breaks up when subjected to stronger mechanical shock. Furthermore it is advantageous if the coating material is dissolved under alkaline conditions, or is readily emulsified by surfactants. This avoids the deposition of undissolved particles or lumps of coating material on the laundry load. This may be important when the  
515 coating material is completely insoluble (for example less than 1 g/l) in water.

As defined herein "substantially insoluble" means having a very low solubility in water. This should be understood to mean having a solubility in water at 25°C of less than 20 g/L, preferably less than 5 g/l, and more preferably less than 1 g/l. Water solubility is measured following the test protocol of ASTM E1148-87  
520 entitled, "Standard Test Method for Measurements of Aqueous Solubility".

Suitable coating materials are fatty acids, adipic acid and C8-C13 dicarboxylic acids, fatty alcohols, diols, esters and ethers. Preferred fatty acids are

those having a carbon chain length of from C12 to C22 and most preferably from C18 to C22. Preferred dicarboxylic acids are adipic acid (C6), suberic acid (C8), azelaic acid (C9), sebacic acid (C10), undecanedioic acid (C11), dodecanedioic acid (C12) and tridecanedioic acid (C13). Preferred fatty alcohols are those having a carbon chain length of from C12 to C22 and most preferably from C14 to C18. Preferred diols are 1,2-octadecanediol and 1,2-hexadecanediol. Preferred esters are tristearin, tripalmitin, methylbehenate, ethylstearate. Preferred ethers are diethyleneglycol mono hexadecylether, diethyleneglycol mono octadecylether, diethyleneglycol mono tetradecylether, phenylether, ethyl naphtyl ether, 2 methoxynaphtalene, beta naphtyl methyl ether and glycerol monooctadecylether. Other preferred coating materials include dimethyl 2,2 propanol, 2 hexadecanol, 2 octadecanone, 2 hexadecanone, 2, 15 hexadecanedione and 2 hydroxybenzyl alcohol. The coating is a hydrophobic material having a melting point preferably of from 40 °C to 180 °C.

In the preferred embodiment, the coating can be applied in a number of ways. Two preferred coating methods are a) coating with a molten material and b) coating with a solution of the material. In a), the coating material is applied at a temperature above its melting point, and solidifies on the tablet. In b), the coating is applied as a solution, the solvent being dried to leave a coherent coating. The substantially insoluble material can be applied to the tablet by, for example, spraying or dipping. Normally when the molten material is sprayed on to the tablet, it will rapidly solidify to form a coherent coating. When tablets are dipped into the molten material and then removed, the rapid cooling again causes rapid solidification of the coating material. Clearly substantially insoluble materials having a melting point below 40 °C are not sufficiently solid at ambient temperatures and it has been found that materials having a melting point above about 180 °C are not practicable to use. Preferably, the materials melt in the range from 60 °C to 160 °C, more preferably from 70 °C to 120 °C.

By "melting point" is meant the temperature at which the material when heated slowly in, for example, a capillary tube becomes a clear liquid. For most



purposes, the coating forms from 1% to 10%, preferably from 1.5% to 5%, of the tablet weight.

555

#### Addition of flow aids

In one embodiment, the process includes adding a flow aid to the particulate detergent composition in a range of from about 0.1% to about 25% by weight of the particulate detergent composition before compaction.

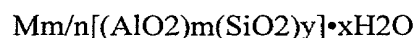
560

As used herein, the term "flow aids" means any material capable of being deposited on to the surface of detergent particles so as to reduce the stickiness of the detergent particles and allow them to flow freely. Flow aids could include porous carrier particles selected from the group consisting of amorphous silicates, crystalline nonlayer silicates, layer silicates, calcium carbonates, calcium/sodium carbonate double salts, sodium carbonates, clays, zeolites, sodalites, alkali metal phosphates, macroporous zeolites, chitin microbeads, carboxyalkylcelluloses, carboxyalkylstarches, cyclodextrins, porous starches and mixtures thereof.

565

The preferred flow aids are zeolite A, zeolite X, zeolite Y, zeolite P, zeolite MAP and mixtures thereof. The term "zeolite" used herein refers to a crystalline aluminosilicate material. The structural formula of a zeolite is based on the crystal unit cell, the smallest unit of structure represented by

570



where n is the valence of the cation M, x is the number of water molecules per unit cell, m and y are the total number of tetrahedra per unit cell, and y/m is 1 to 100.

Most preferably, y/m is 1 to 5. The cation M can be Group IA and Group IIA elements, such as sodium, potassium, magnesium, and calcium.

575

In the preferred embodiment of the present invention, the flow aid is added in an amount in a range, desirably, from about 0.1% to about 25% by weight of the particulate detergent, more desirably from about 1% to about 15% by weight, preferably from about 1% to about 10% by weight, and most preferably in an amount of about 5% by weight. It is undesirable to add more than 25% by weight of the flow aid because too excessive a force would be needed to make the detergent particles to stick together and stay in a particulate form. Flow aid addition in an amount less than about 0.1% by weight is also undesirable because

580

little or no reduction in the stickiness of the detergent particles would occur, which  
585 upon compression into a particulate form would cause the resultant detergent  
tablet to not disintegrate readily when placed in water in a washing machine.

In one embodiment, the flow aids have a perfume adsorbed on their surface  
before being deposited on the detergent particles. Preferably, the flow aids are  
zeolites preferably containing less than about 20% desorbable water, more  
590 preferably less than about 8% desorbable water, and most preferably less than  
about 5% desorbable water. Such materials may be obtained by first  
activating/dehydrating by heating to about 150 to 350 C, optionally with reduced  
pressure (from about 0.001 to about 20 Torr). After activation, the perfume is  
slowly and thoroughly mixed with the activated zeolite and, optionally, heated to  
595 about 60°C for up to about 2 hours to accelerate absorption equilibrium within the  
zeolite particles. The perfume/zeolite mixture is then cooled to room temperature  
and is in the form of a free-flowing powder. The term "perfume" is used to  
indicate any odoriferous material which is subsequently released into the aqueous  
bath and/or onto fabrics contacted therewith. The perfume will most often be  
600 liquid at ambient temperatures. A wide variety of chemicals are known for  
perfume uses, including materials such as aldehydes, ketones and esters. More  
commonly, naturally occurring plant and animal oils and exudates comprising  
complex mixtures of various chemical components are known for use as perfumes.  
The perfumes herein can be relatively simple in their compositions or can  
605 comprise highly sophisticated complex mixtures of natural and synthetic chemical  
components, all chosen to provide any desired odor. Typical perfumes can  
comprise, for example, woody/earthy bases containing exotic materials such as  
sandalwood, civet and patchouli oil. The perfumes can be of a light floral  
fragrance, e.g., rose extract, violet extract, and lilac. The perfumes can also be  
610 formulated to provide desirable fruity odors, e.g., lime, lemon, and orange. Any  
chemically compatible material which exudes a pleasant or otherwise desirable  
odor can be used in the perfumed compositions herein. Perfumes also include pro-  
fragrances such as acetal pro-fragrances, ketal pro-fragrances, ester pro-fragrances  
(e.g., digeranyl succinate), hydrolyzable inorganic-organic pro-fragrances, and

615 mixtures thereof. These pro-fragrances may release the perfume material as a result of simple hydrolysis, or may be pH-change-triggered pro-fragrances (e.g., pH drop) or may be enzymatically releasable pro-fragrances.

In the preferred embodiment, the amount of perfume adsorbed on the carrier material, such as zeolite for example, is preferably in the range of about 0.1% to about 50% by weight, more preferably in the range of about 0.5% to about 25% by weight, and most preferably in the range of about 1% to about 15% by weight of zeolite powder.

#### Compaction of particulate detergent to form non-particulate detergent product

In the preferred embodiment, the process still further includes the step of compacting the particulate detergent composition having at least 10% reduced porosity by applying a pressure in an amount sufficient to form the water-dispersible, non-particulate detergent product having a density of at least about 1000 g/l. It is desirable to form a detergent tablet that has a density of at least about 1000 g/l so that the tablet will sink in water. If the density of the detergent tablet is less than about 1000 g/l, the tablet will float when placed in the water in a washing machine and this will detrimentally reduce the dissolution rate of the tablet in the water. It is desirable to apply at least that much pressure as is sufficient to compress the particulate detergent material to form a tablet having a density of at least about 1000 g/l. Too little a pressure will result in a compressed tablet with a density less than about 1000 g/l.

#### EXAMPLE A

Detergent tablets are formed from detergent particles having at least 10% reduced porosity before such particles are compressed into a tablet form, according to the following composition:

640

Table A.1

<u>Ingredients</u>	<u>% by weight</u>
Detergent particles	95.00
polyethylene glycol (MW=200)	5.00
<hr/>	
Total	100.00

645

The detergent particles have the following composition:

Table A.2

	<u>Particulate detergent Ingredients</u>	<u>% by weight</u>
	C <sub>12-16</sub> linear alkylbenzene sulfonate	8.80
650	C <sub>14-15</sub> alkyl sulfate/C <sub>14-15</sub> alkyl ethoxy sulfate	8.31
	C <sub>12-13</sub> alkyl ethoxylate	1.76
	polyacrylate (MW=4500)	2.40
	polyethylene glycol (MW=4000)	0.96
	sodium sulfate	8.40
655	aluminosilicate	21.28
	sodium carbonate	16.80
	protease enzyme	0.32
	sodium perborate monohydrate	2.08
	lipase enzyme	0.17
660	cellulase enzyme	0.08
	NOBS extrudate	4.80
	citric acid monohydrate	2.25
	sodium bicarbonate	2.75
	sodium acetate	15.00
665	free water	1.60
	other minor ingredients (perfume etc.)	2.24
<hr/>		
	Total	100.00

670 The detergent tablet formed is coated with a coating according to the following composition:

Table A.3

	<u>Ingredient</u>	<u>% by weight</u>
	Detergent tablet having PEG	91.10
675	Coating:	
	dodecanedioic acid	8.00

carboxymethyl cellulose	0.90
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Total	100.00
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680        Optionally, a flow aid (zeolite) is also added to the particulate detergent composition in about 5% by weight of the detergent and mixed by one of various methods, such as agitation for example.

685        The tablets are formed by compressing the tablet ingredients in a cylindrical die having a diameter of 55 mm using a laboratory press having a trade name Carver Model 3912, to form a tablet having a height of 20 mm. The formed tablets were then coated with the protective coating by dipping the tablet into a molten bath of the coating for about 3 seconds. The molten coating bath is maintained at a temperature of about 145 degrees centigrade.

690        The term "NOBS extrudate" as used herein, is an acronym for the chemical sodium nonanoyloxybenzene sulfonate, commercially available from Eastman Chemicals, Inc. The carboxymethyl cellulose used in the above example is commercially available from Metsa-Serla and sold under the trade name, Nymcel ZSB-16.

#### Test for determining dispersibility in water

695        The following method is used to measure the rate of dispersion (ROD) of a detergent tablet expressed as percentage residue remaining after "t" minutes, where "t" is 3, 5 and 10 minutes. The equipment used includes a 5000 ml glass beaker, a stopwatch with alarm, an electrical stirrer with variable speed IKA RW 20 DZM or equivalent, a cage made of perforated metal gauze (diameter 52 mm, height 40 mm  
700        having 16 apertures each of about 2.5 mm square) and a weigh scale with accuracy of 0.1 grams.

705        The method includes the following steps: The beaker is filled with 4000 ml (+/-50 ml) of distilled water at 20 °C (+/- 1 °C ). The cage tester is mounted in the electrical stirrer. A tablet with a known weight is placed in the cage and the cage is connected to the stirrer. The cage is submerged in the water with the cage suspended about half way down the beaker and the stirrer is started at a fixed speed of 80 rpm. The stopwatch is started. The stirrer is stopped after 3 minutes. The

cage is lifted out of the water and the tablet residue remaining in the cage is weighed. The % residue is calculated with the following equation:

$$\% \text{ residue} = \frac{\text{Tablet weight after "t" minutes}}{\text{Initial tablet weight}} \times 100$$

The remaining tablet is placed back in the cage and the process is repeated for an additional 2 and 5 minutes to give yield data for tablet dispersion after 3, 5 and 10 minutes.

As used herein, the term "dispersibility in water" is defined as a measure of the % residue, as calculated above, after 3 minutes. In other words, for example, a detergent tablet which has 5% by weight less residue than another detergent tablet would be deemed to have 5% greater dispersibility in water.

It is unexpectedly and surprisingly discovered that the non-particulate detergent product, e.g., a detergent tablet, has at least about 5% greater dispersability in water as compared to another non-particulate detergent product having a density of at least about 1000 g/l but not having nonionic liquid added to the detergent composition according to this invention.

In another embodiment of the present invention, a method of laundering fabric materials in a washing machine includes the steps of providing a flexible porous bag adapted for receiving a non-particulate detergent product, providing a non-particulate detergent product, placing the non-particulate detergent product within the flexible porous bag, and placing the flexible porous bag containing the detergent product in the washing machine with the fabric materials to be washed.

The flexible porous bag is permeable to water and to the washing medium and is thus adapted for permitting entry of an aqueous washing medium through the bag, thereby dissolving the non-particulate detergent product placed therein, into the aqueous washing medium, and releasing a resultant wash solution from inside of the bag to outside of the bag and into the aqueous wash medium during a wash cycle.

The flexible porous bag is made of a material capable of retaining the non-particulate detergent product without allowing it to pass through until the detergent

product has dissolved in the washing medium. The bag is also made of a material capable of withstanding the temperatures of washing laundry in a washing machine.

740 The process of the invention may be applied not only to non-particulate detergents but also to any non-particulate product which is active during washing, such as, for example, bleaching agents, such as agents releasing chlorine or active oxygen (peroxygen compounds), bleaching catalysts, bleaching activators, bactericides, foam regulators, whiteners, agents preventing the re-deposition of soil, enzymes, 745 softeners, agents capable of removing grease stains or other constituents having no direct effect on the soiling but capable of taking part in the laundry washing process.

The flexible bag may be made from any material which offers a sufficient resistance to water, such as a woven or non-woven material produced from natural or synthetic fibers. For example, the bag is formed of pure cotton either in the form of 750 a fabric with a mesh opening of less than about 0.5 mm or in the form of a non-woven article with openings having a size in a range of from about 0.5 mm to about 0.8 mm.

Accordingly, having thus described the invention in detail, it will be obvious to those skilled in the art that various changes may be made without departing from 755 the scope of the invention and the invention is not to be considered limited to what is described in the specification.

## WHAT IS CLAIMED IS:

1. A process for producing a water-dispersible, non-particulate detergent product from low density detergent particles, characterized by the steps of:
  - (a) providing low density detergent particles having an intra-particle porosity in a range of from 5% to 90% by volume;
  - (b) adding a liquid to said low density detergent particles in an amount sufficient to reduce said intra-particle porosity by at least 10%; and
  - (c) compacting said low density detergent particles having said reduced intra-particle porosity, by applying a pressure in an amount sufficient to form said water-dispersible, non-particulate detergent product having a density of at least 1000 g/l.
2. The process of claim 1 wherein said low density detergent particles of step (a) have an intra-particle porosity in a range of from 10% to 80% by volume.
3. The process of any of claims 1-2 wherein said low density detergent particles of step (a) have a bulk density no less than 400 g/l.
4. The process of any of claims 1-3 wherein said low density detergent particles of step (a) have a bulk density in a range of from 400g/l to 850g/l.
5. The process of any of claims 1-4 wherein said liquid is a nonionic surfactant and is added to said low density detergent particles in an amount sufficient to reduce said intra-particle porosity by at least 20%.
6. The process of any of claims 1-5 wherein said liquid is added in an amount in a range of from 0.1% to 10% by weight of said low density detergent particles.
7. The process of any of claims 1-6 wherein said liquid is selected from the group consisting of nonionic surfactants, anionic surfactants, water, polyethylene glycol, and mixtures thereof.



8. The process of any of claims 1-7 wherein said liquid is a nonionic surfactant.
9. The process of any of claims 1-8 wherein said liquid is polyethylene glycol having a molecular weight less than 1000.
10. The process of any of claims 1-9 wherein said water-dispersible, non-particulate detergent product has at least 5% greater dispersability in water as compared to a non-particulate detergent product having a density of at least 1000 g/l but not having said liquid added to said low density particulate detergent composition per step (b).
11. A rapidly water-dispersible non-particulate detergent product formed by a process characterized by the steps of:
  - (a) providing low density detergent particles having an intra-particle porosity in a range of from 5% to 90% by volume;
  - (b) adding a liquid to said low density detergent particles in an amount sufficient to reduce said intra-particle porosity by at least 10%; and
  - (c) compacting said low density detergent particles having said reduced intra-particle porosity, by applying a pressure in an amount sufficient to form said water-dispersible, non-particulate detergent product having a density of at least 1000 g/l.
12. A method of laundering soiled clothes characterized by the step of immersing said soiled clothes in an aqueous medium containing an effective amount of a non-particulate detergent product made by a process according to any of claims 1-11.
13. A process for producing a water-dispersible, non-particulate detergent product from a low density detergent composition, characterized by the steps of:
  - (a) providing spray dried detergent granules having a bulk density less than 650 g/l;

(b) providing agglomerated detergent particles having a bulk density in a range of from 700 g/l to 900 g/l;

(c) mixing said spray dried detergent granules and said agglomerated detergent particles to form a low density detergent composition, wherein said spray dried detergent granules are present in a range of from 40% to 80% by weight of said low density detergent composition and said agglomerated detergent particles are present in a range of from 20% to 60% by weight of said low density detergent composition;

(d) adding a liquid to said low density detergent composition in an amount sufficient to reduce said intra-particle porosity by at least 10%; and

(e) compacting said low density detergent composition having said reduced intra-particle porosity, by applying a pressure in an amount sufficient to form said water-dispersible, non-particulate detergent product having a density of at least 1000 g/l.

14. A method of laundering fabric materials in a washing machine, characterized by the steps of:

providing a flexible porous bag adapted for receiving a non-particulate detergent product;

providing a non-particulate detergent product made by a process according to any of claims 1-13;

placing said non-particulate detergent product within said flexible porous bag;

placing said flexible porous bag containing said detergent product in said washing machine with said fabric materials to be washed; and

said flexible porous bag being adapted for permitting entry of an aqueous washing medium through said bag, thereby dissolving said non-particulate detergent product placed therein, into said aqueous washing medium, and releasing a resultant wash solution from inside of said bag to outside of said bag into said aqueous wash medium during a wash cycle.

## DECLARATION COMBINED WITH POWER OF ATTORNEY

Page 1 of 2

Attorney Docket No. 7129

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled **PROCESS FOR MAKING A READILY WATER-DISPERSIBLE NON-PARTICULATE DETERGENT PRODUCT FROM LOW-DENSITY PARTICULATE DETERGENT COMPOSITION** the specification of which

☐ is attached hereto.  
☒ was filed on April 21, 1999 as United States Application No. or  
 PCT International Application Number PCT/IB99/00708  
 and was amended on (if applicable)

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in Title 37 Code of Federal Regulations §1.56.

I hereby claim foreign priority benefits under Title 35 United States Code §119(a)-(d) or §365(b) of any foreign application(s) for patent or inventor's certificate, or §365(a) of any PCT International application which designated at least one country other than the United States of America, listed below and have also identified below any foreign application for patent or inventor's certificate, or of any PCT international application having a filing date before that of the application on which priority is claimed:

<u>Prior Foreign Application(s)</u>			<u>Priority Claimed</u>	
<u>(Number)</u>	<u>(Country)</u>	<u>(Day/Month/Year Filed)</u>	<input type="checkbox"/> Yes	<input type="checkbox"/> No

I hereby claim the benefit under Title 35, United States Code §119(e) of any United States provisional application(s) listed below.

<u>Application Serial No.</u>	<u>Filing Date</u>	<u>Application Serial No.</u>	<u>Filing Date</u>
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I hereby claim the benefit under Title 35, United States Code §120 of any United States application(s), or §365(c) of any PCT International application designating the United States of America, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of Title 35, United States Code §112, I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations §1.56 which became available between the filing date of the prior application and the national or PCT international filing date of this application:

U.S. Parent Application Number	PCT Parent Number	Parent Filing Date (MM/DD/YYYY)	Parent Patent Number (If applicable)

As named inventor, I hereby appoint the following registered practitioner(s) to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith:

<u>Attorney Name</u>	<u>Attorney Reg Number</u>
Brian M. Bolam	<u>37,513</u>
Jacobus C. Rasser	<u>37,043</u>
Kim W. Zerby	<u>32,323</u>
S. Robert Chuey	<u>39,140</u>
Pankaj M. Khosla	<u>37,978</u>
T. David Reed	<u>32,931</u>
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<u>Ivorydale Technical Center, 5299 Spring Grove Avenue, Cincinnati, Ohio 45217</u>	

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Full name of sole or first joint inventor Adrian John Waynforth Angell

Inventor's signature *Adrian Angell* 5/24/99  
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Full name of fourth joint inventor, if any \_\_\_\_\_

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## DECLARATION COMBINED WITH POWER OF ATTORNEY

Page 1 of 2  
Attorney Docket No. 7129

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☐ is attached hereto.  
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 and was amended on (if applicable)

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<u>Prior Foreign Application(s)</u>			<u>Priority Claimed</u>	
<u>(Number)</u>	<u>(Country)</u>	<u>(Day/Month/Year Filed)</u>	<input type="checkbox"/> Yes	<input type="checkbox"/> No

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As named inventor, I hereby appoint the following registered practitioner(s) to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith:

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S. Robert Chuey	39,140
Pankaj M. Khosla	37,978
T. David Reed	32,931
Timothy B. Guffey	41,048

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Full name of sole or first joint inventor Adrian John Waynforth Angell  
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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In the matter of

U. S. National Phase Entry :  
Under 35 USC 371 from :  
the International Application of :  
ANGELL, Adrian J ohn Waynforth et al :  
Int'l Application No. PCT/IB99/00708 :  
Filed in the RO/US on 21 April 1999 :  
Entitled: Water-Dispersible Non-Particulate :  
Detergent Product from Low-Density :  
Particulate Detergent Particles :

ASSOCIATE POWER OF ATTORNEY

Assistant Commissioner for Patents  
Box PCT  
Washington, D.C. 20231


Dear Sir:

You are requested to recognize B. M. Bolam (Registration No. 37,513); K. W. Zerby (Registration No. 32,323); J. J. Camp (Registration No. 44,582); F. C. Turner (Registration No. 39,863); C. B. Cook (Registration No. 39,151); M. Dressman (Registration No. 42,498); R. S. Echler, Sr. (Registration No. 41,006); and I. S. Robinson (Registration No. 43,348) of The Procter & Gamble Company, Cincinnati, Ohio, as Associate Attorney to prosecute this application, to make alterations and amendments therein, and to transact all business in the Patent Office connected with the application or with the patent granted thereupon.

Please address all future communications to:

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Respectfully submitted for Applicants,

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